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Industrial Crops and Products 21 (2005) 379–385

INDUSTRIAL CROPS AND PRODUCTS
AN INTERNATIONAL JOURNAL

www.elsevier.com/locate/indcrop

Improving particle separation from an ethanol extract to water: settling dependence on fine particle content☆

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Received 23 February 2004; accepted 10 June 2004

Abstract

Removing solid particles from ethanolic corn extracts by gravitational settling into a water layer has been studied as part of a project to develop a low cost method to extract ethanol-soluble protein from corn meal. Settling has several advantages over industrially proven methods of liquid/particle separation: (1) less expensive equipment; (2) particles that settle at different rates can be collected from different outlets, and (3) extract liquid entrained by settling particles dissolves in the water from which the ethanol can be recovered by distillation. When fine particles were pumped as part of the extract into the 5-l settling layer at a high enough rate, they formed a particle layer at the extract/water interface. The particle layer prevents further settling and is a non-sustainable operating condition. The layer does not form when the mass fraction of particles in the extract was 0.11 or less. This value is greater than the value obtained by extrapolating published settling data for denser (mineral) particles. Overall, entrainment of extract liquid for the settling was about the same as entrainment during centrifugation. In-line measurements and recordings of ethanol concentration, density, and particle content of the water stream carrying settled coarse particles from the settling tank showed that specific entrainment was greater for the finer particles.
Published by Elsevier B.V.

Keywords: Sedimentation; Corn extract; Interface; Entrainment

1. Introduction

A project to develop an inexpensive process to separate ethanol-soluble corn protein from corn meal led to a study of various ways of separating the extracted corn particles from the extract liquid (Dickey et al., 2002). It was found that displacement of the extract liquid with water or an ethanol solution from a stationary bed of the settled extract was too slow to be practical. Decanter centrifugation of the extract seemed to be workable, but the dry product stream contained 45% liquid from which the ethanol would be relatively expensive to recover. Gravitational settling of the extracted corn particle from a thin layer of extract flowing over 1000 l of water resulted in low loss of extract liquid with the settled particles, about the same as that

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0926-6690/$ – see front matter. Published by Elsevier B.V.
doi:10.1016/j.indcrop.2004.06.002

left on the centrifuged particle product. The viability of settling depends on minimizing the amount of extract liquid entrained by the particles and carried with them to the water. Also, water must be pumped through the bottom of the tank at a velocity high enough to move the settled particles to the tank outlet, but not so high that it causes convective mixing between the water and extract. Settling tests in a 35-l tank showed that the base of the tank must be inclined at 35° from horizontal and the water flow kept close to the base to keep particles from accumulating at the bottom of the tank (Dickey et al., 2003). Based on these tests, use of sedimentation instead of centrifugation was estimated to reduce the capital cost of a zein recovery plant by 50%. Further tests indicated that the capacity of a settling tank was limited because of the formation of a fine particle layer at the extract/water interface.

Numerous reports on sedimentation (Bustos et al., 1999; Buerger, 2000; Buerger et al., 2000) and gravity current (Haertel and Meiburg, 1999) modeling are available. The purpose of sedimentation in a continuous thickener differs from the settling method being developed because entrainment is not of interest in thickeners. Compression of particles at the bottom of the thickener is desirable because it provides a convenient form for discharge, whereas the best condition for settling is where the particles fall separately.

The major variable in all sedimentation models is the local particle volume fraction. To prevent hindered settling, and in the case of a liquid/liquid interface, formation of a dense consolidated layer that will retard the descent of the particles, the local particle volume fraction must be kept below a critical value. At this critical volume fraction, a suspension will no longer resist compression elastically and will irreversibly consolidate (Landman et al., 1988). Numerical models of published experimental data (Garrido et al., 2000) were used to estimate the critical volume fractions for mixtures of minerals in various liquids. The critical fraction should decrease with decreasing density difference because lower net gravitational force on the particles causes slower separation of the particles and faster accumulation. The density differences (DD) of the four experiments (Garrido et al., 2000) with identified critical fractions (CF) and the origin varied linearly with critical fraction as

$$\mathrm{CF} = 4 \times 10^{-5}\mathrm{DD} - 0.0075, \qquad R^2 = 0.93.$$

We measured the extract liquid specific gravity of corn meal extracts (0.87). The specific gravity of the most rapidly settling particles is 1.4, and therefore, the maximum DD is about 530 kg/m^3, one-third to one-eighth of the DDs for the published mixtures. By using the corn meal extract and DD in the equation to calculate a critical volume fraction for the corn meal extraction mixture, the result indicates that the critical fraction is ∼0.014, or about one-tenth of the particle fraction of our extracts. From the standpoint of extraction efficiency and minimum cost, extraction with the maximum mass of corn meal per mass of extracting liquid (highest particle fraction) is preferable. Previous extractions were carried out with this maximum corn meal fraction (0.13), determined by the pump and extraction tank agitation system. In settling an extract with this particle volume fraction, clumps of particles would occasionally clog the peristaltic pump and tubing used to feed the settling tank. Both the length of the tank and the depth of the extract layer are important for the settling process unlike the one-dimensional settling models published where only the depth is important. In polydisperse mixtures, the larger particles can entrap smaller ones during settling (Bustos et al., 1999). Furthermore, larger particles settle quickly and retain less extract liquid per particle mass than the smaller ones so that the settling in the downstream section of the tank has a lower particle fraction than the upstream section where extract is fed to the tank.

When the extract was fed to the (top layer) of the settling tank, the larger particles descended quickly to the water and were carried out of the tank with the water stream leaving from the same end of the tank they entered. Finer particles were carried downstream with the extract liquid and settled and were removed with the water stream leaving from the other end of the tank. Very fine particles that did not settle to the water were carried out with the settled extract liquid. At a given extract feed rate, the possibility for solid particle layer formation at the liquid/liquid interface will depend on the fine particle content of the extract. Formation of a particle layer is undesirable because it continues to thicken, preventing settling of particles of increasingly coarser size. Once a layer begins to form, it usually grows until the settling tank is clogged. The goal of this investigation was to determine the maximum rate of fine particles that could be settled without forming a layer of particles at the water/extract interface.

2. Experiment

2.1. Extraction of corn meal

Typically, 9.1 kg of corn meal (reduced to a median size of 0.35 mm as determined by sieve separation) was mixed in a tank for 90 min at 50 °C with 90 kg of 70% ethanol solution. One batch of corn meal was extracted at 39 °C.

The slurry was agitated by two scraper blades attached to a central shaft rotating at 10 rpm and circulated out of the tank through a centrifugal pump (Fristram, model FP702, Middletown, WI) at 40 kg/min and back into the tank to increase the disruption of the corn particles.

2.2. Settling of extract

After extraction, the extract was cooled to ambient temperature and pumped into the settling tank, shown in Fig. 1, using a Masterflex L/S pump (Cole–Parmer Instrument Co., Vernon Hills, IL) running at either 100 or 50 rpm through size 36 Tygon® tubing to the right side of the settling tank at 0.44 or 0.22 kg/min. Settled extract (liquid) was drawn from the left side using an identical tubing pump continuously running at 150 rpm, thereby, maintaining the extract liquid/air interface at the outlet port height.

The settling tank used was a slightly modified version of one described previously (Dickey et al., 2003). Transparent settling tank walls enabled observation of the interface and the movement of solid particles through it. As shown in Fig. 1, the tank has three bases inclined 35° from the horizontal that direct the settling particles to the water outlet ports. In-flowing water was directed along the bases to confine the momentum to the lowest part of the particle containing water volume. The stagnant water below the bases was separated from the water flowing downward above the bases. Earlier testing had shown that water flow along bases with less inclination is unable to keep settling particles from collecting at the bottom of the settling tank.

Just prior to pumping the extract, a 5–6 kg layer of 70% ethanol was pumped onto the water to form a zein-free ethanol solution layer above the water. The zein dissolved in the 70% ethanol extract solution will precipitate when the ethanol concentration drops to 40%, therefore, some protein precipitation is unavoidable, but it can be reduced by the initial protein-free layer.

Solid particles settled from the extract layer as it flowed across the top of the settling tank. They were collected and transferred from the settling tank by two circulating water streams. Finer and more slowly settling particles descended to the larger region on the extract liquid outlet side. Coarse particles settled rapidly to the bottom of the tank on the extract feed end. Each stream flowed out of each end and through solids collecting vessels (not shown) and back to ports in the

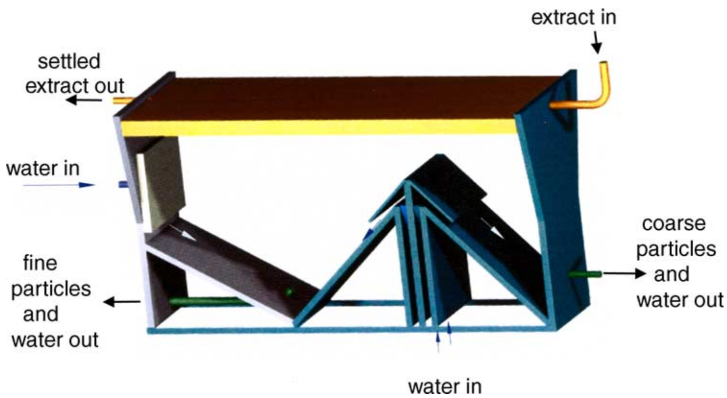

Fig. 1. Settling tank, with flow direction of extract, fine particle-transporting water, and coarse particle transporting water.

settling tank. Two dual headed Masterflex L/S pumps using size 73 tubing (Cole–Parmer Instrument Co., Vernon Hills, IL) were used to pump water out of, and back into, each side of the settling tank. The tubing compression of each of the pumps, running at 400 rpm, was adjusted so that the water level in the settling tank remained constant, several cm below the outlet port. Because the top of the extract layer was fixed at the height of the outlet port, the height of the water level controlled the thickness of the extract layer. The output stream from the right side was pumped to a piping loop shown in Fig. 2, containing a model 8100 in-line ultrasonic probe (Rhosonics, Baarn, Netherlands) that enabled measurement of ethanol content by measuring the sound velocity of the stream. The loop also contained a Masterflex I/P pump using size 92 tubing (Cole–Parmer Instrument Co.) that was installed to generate sufficient flow rate (24 kg/min) to keep solids from collecting in the probe. The probe was mounted with the long axis vertical to insure that solids would not accumulate; the loop including the probe contained 3.5 l. Water leaving the ultrasonic probe loop flowed to a tank from which it was pumped to an in-line coriolis sensor (MicroMotion, model CM050, Boulder, CO) using another I/P pump. The coriolis meter enabled measurement of density, temperature, and flow rate.

The water stream leaving the coriolis sensor flowed to a 15.2 cm hydrocyclone (Centri-cleaner, Sprout Bauer, Muncy, PA) with an underflow outlet diameter of 0.635 cm. Particles from the underflow outlet were

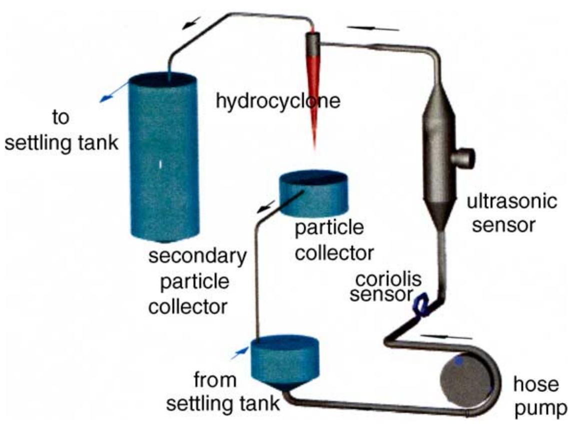

Fig. 2. Coarse particle transporting water is injected into a collection loop and overflow from the loop is returned to the settling tank.

collected in a tank containing a weir and most of the overflowing liquid was returned to the measurement loop. A stream of water at a flow rate slightly higher than that flowing into the measurement loop was returned to the settling tank.

During the settling, which took from 5 to 6 h, samples were taken when about 14 kg of settled extract liquid had been collected in a pail and transferred to a storage drum. When all of the extract that could be pumped from the extraction tank had been removed, the remaining material in the tank was rinsed out with a known amount of water, the specific gravity of the mixture and of the liquid phase measured using an electronic floor scale and hydrometers. This allowed determination of the ethanol and solid particle content of the material left in the extraction tank. The ethanol and particle contents of the settling tank, and tanks containing the water circulated to and from each side of the settling tank were measured in the same way. Samples of the extract liquid product were taken and analyzed later for ethanol, lipid, protein, and solid content. For each vessel the solid fraction, x, and an effective corn particle specific gravity, y, were obtained from the equation:

$$x/y + (1 - x)/(\text{liquid specific gravity}) = 1/(\text{mixture specific gravity}) \quad (1)$$

The equation is based on adding the volume of the corn particles and the volume of the liquid to get the mixture specific gravity that was measured along with the liquid specific gravity. The vessel equation was used to determine the particle mass in each vessel (in terms of y) and then all of these were set equal to the original corn mass to determine y, which varied from 1.33 to 1.55.

3. Results

In a typical run, 99.1 kg of 9.2 wt.% (0.059 volume fraction) corn meal extract was settled in 5 h. No solid particle layer formed in the settling tank. Eighty kilograms of extract liquid was collected and transferred to the storage drum along with 6 kg from the top of the settling tank. Five samples of the settled extract liquid were analyzed. They had the average composition: dissolved solids, 1.0; including lipid, 0.16; protein, 0.34;

Table 1
Particle layer formation and entrainment for settling corn meal extracts

Mass ratio and formation of particle layer						Mass ratio/entrainment					Fine particle transit rates and rate ratios		
Run number	Extract mass ratio[a], corn meal/liquid	Extract volume ratio[a], corn meal/liquid	Interfacial particle layer formed	Particles in settling tank at the end of run (kg)	Corn specific gravity[b]	Entrained mass ratio[c], coarse	Entrained mass ratio[c], fines	Overall entrained mass ratio[d], extract/particles	Recovered mass ratio[e], coarse/fine	Extract mass ratio[f], extract liquid/fines	Residence time in extract layer[g] (min)	Average transit rate[h] ($kg/h\text{-}m^2$)	Transit rate/settling rate[i]
5	0.10	0.062	No	0.68	1.40	0.97	1.17	1.05	0.93	2.2	36.5	12.3	0.91
7	0.12	0.075	No	0.9	1.55	0.37	3.78	0.75	0.12	6.8	23.5	2.9	0.68
9	0.12	0.077	No	0.1[j]	1.33	0.92	1.06	0.97	0.50	2.9	10.7	13.5	0.98
8	0.13	0.081	Yes	3.0	1.40	–	–	–	–	–	14.6	8.8	0.64
6	0.15	0.093	Yes	1.14	1.45	–	–	–	–	–	36.2	1.9	0.55

[a] The solid and liquid remaining in the extraction tank after settling was subtracted from the initial solids and liquid in the extraction tank before calculating the ratio of the settled extract.
[b] Calculated using Eq. (1) followed by setting the sum of the solid mass for each vessel equal to the original corn mass.
[c] The ratio was determined by dividing the extract liquid mass transferred to the water by the solid particle mass that was needed to give the final measured ethanol and solid content in the water.
[d] The ratio is the mass of all extract liquid transferred divided by the mass of all particles recovered.
[e] The ratio is the mass of the solid particles recovered from the extract outlet end (of the settling tank) divided by the mass of particles recovered from the inlet end.
[f] The ratio is the mass of all the extract liquid transferred divided by the mass of the fine particles recovered (from outlet end).
[g] Estimated by dividing the extract layer mass, 4.5 kg, by the average extract feed rate.
[h] Calculated by dividing the mass of recovered fine particles by the time of the separation and the cross section (area) of the fine particle collection (at the interface), 568 cm^2.
[i] The transit rate (in the preceding column) was divided by the sum of the particle mass accumulated at the layer, two-thirds of the values in column 5, and the (fine) particles recovered.
[j] Determined by difference, using all the other solid determinations, and the original corn meal mass.

and ethanol, 60.9 wt.%. The fine particles recovered from the collection pails were less than 0.1 wt.%.

The ultrasonic probe indicated a steady increase of the ethanol content in the water stream from 0 wt.% at the beginning to 2.5 wt.% by the end of the run; 8.2 kg of solids were fed into the settling tank over this period. The overall rate of ethanol content increase was 0.0085 wt.%/min. The ethanol content increase did not change when the extract feed rate was lowered from 100 to 50 rpm after 120 min. In the tanks connected to the feed side of the settling tank, 5.5 kg of solid particles were recovered in 99 kg of 2.5% ethanol solution. On the extract outlet side (finer particles), 2.5 kg of particles were recovered in 100 kg of 2.7% ethanol solution.

When a 13 wt.% (0.0837 volume fraction) extract was settled, an interfacial particle layer formed after 2 h and grew to fill the settling tank after 5 h. When an extract containing 11.5 wt.% corn meal (0.075 vol.%) was settled, a thin, 1 cm layer formed, but did not increase in thickness. The mass of particles in the settling tank at the end of a run varied directly with the extract solids content; the particle mass in the tank reflects the extent to which particles were collecting at the interface and the solids extract content the mass of particles being settled in about the same amount of time. Average residence time for the extract liquid in the settling tank and very fine particles it retained was estimated by dividing the extract layer mass of 4.5 kg, determined by multiplying the measured extract density by the volume of the layer, by the average extract feed rate. A 1 kg or larger mass of particles on the bottom of the tank at the end corresponds to a definite visible particle layer at the interface. Smaller final amounts are particles that would have been cleared if the run had not been stopped.

3.1. Formation of a particle layer

Based on the run results included in the first six columns of Table 1, the particle volume fraction of an ethanol/corn meal extract can be as great as 0.07 (mass fraction up to 0.107) and avoid formation of a particle layer at the interface. This critical mass fraction is higher than expected from the published settling results for denser particles. The behavior may be partly due to the heterogeneity of the extracted corn meal, the larger particles falling quickly to the water, thereby, reducing the number of suspended particles in the extract layer whose descent rate is slow enough to collect in the time available. The result is unexpected, in that, a layer could have formed because of the precipitation of zein and/or lipid from the extract; apparently, no precipitates formed, or if they did, they did not impede settling through the interface. We postulate that a corn lipid layer may form between the water and extract which would prevent zein precipitation and the relatively high mass fraction of particles that can transit through the interface is consistent with formation of a fluid lipid layer and not a zein film.

3.2. Entrainment ratio dependence on particle size

The entrainment ratio was calculated separately for the extract feed side of the tank (coarse, faster settling particles) and the outlet side (fines), using the measurements of particle and ethanol content in the tanks connected to the settling tank. By using data from runs in which a layer was not judged to have formed, shown in columns 7–11 of Table 1, the entrainment ratios were higher for the fine particles than for the coarse particles. The run 7 numbers are unlike the other two as a result of a higher fraction of larger particles, which were the result of a lower extraction temperature of 39 °C.

To reduce the entrainment overall, it would suffice to reduce the amount of fine particles in the extract. However, to dissolve the zein from the corn meal, it is necessary to reduce the endosperm structure to the starch granule size to expose the zein-containing bodies to the ethanol solution. Thus, the particle size control options are limited to a minimum size of around 50 μm, an average size for starch granules.

The data in columns 12–15 of Table 1 suggest that when the particles move through the interface at a rate two-thirds or more of the rate of (fine) particles settling to it a visible layer will not be formed. However, for a long settling run and stable (non-growing) interfacial layer, the rates would necessarily be equal. This apparent contradiction is a result of the particle layer (sometimes thin) accumulating at the start of the run and the particle mass included in its formation is a deceptively high fraction of the fine particle mass settled in a short run.

4. Conclusions

We showed previously that corn meal extracts could be settled and the particles removed by circulating water with modest entrainment, at a rate of 12 kg/h m^2 when the water flow rate was 30 times the extract feed rate, in the tank used in this work (Dickey et al., 2003). We subsequently describe the investigation of another limit to extract settling, the formation of a particle layer at the interface. The data in columns 1–6 of Table 1 show that an extract containing less than 10 wt.% corn did not form a particle layer at the interface.

Settling can be used for particle/liquid separations of other agricultural extracts and recovery of solvents or liquid containing valuable solutes, such as enzymes, which would be more readily recovered or reused undiluted. Solvent conservation is increasingly important in operation of large scale processing facilities and avoiding the need to recover solvent by evaporation from a solids mass (the centrifuge dry product) is the primary benefit of settling. Saving the capital and operating expense of a solvent-capable centrifuge is a significant secondary advantage of settling a solvent/particle mixture. The final advantage, which has not yet been explored, is that the particles can be classified during settling.

Acknowledgements

The authors thank Michael F. Dallmer who modified and operated the equipment to produce data on which this report is based and Mike Kurantz for performing the analytical measurements.

References

Buerger, R., 2000. Phenomenological foundation and mathematical theory of sedimentation-consolidation processes. Chem. Eng. J. 80, 177–188.

Buerger, R., Concha, F., Tiller, F.M., 2000. Applications of the phenomenological theory to several published experimental cases of sedimentation processes. Chem. Eng. J. 80, 105–115.

Bustos, M.C., Concha, F., Buerger, R., Tory, E.M., 1999. Sedimentation and thickening. Phenomenological foundation and mathematical theory. In: Mathematical Modeling: Theory and Applications, vol. 8. Kluwer Academic Publishers, Dodrecht, The Netherlands.

Dickey, L.C., Parris, N., Craig, J.C., Kurantz, M.J., 2002. Separation of maize particles from alcohol extracts with minimal losses. Ind. Crops Prod. 16, 145–154.

Dickey, L.C., McAloon, A., Parris, N., 2003. Minimizing entrainment of extract liquid by settling maize particles. Ind. Crops Prod. 18, 77–84.

Garrido, P., Buerger, R., Concha, F., 2000. Settling velocities of particulate systems. 11. Comparison of the phenomenological sedimentation-consolidation model with published experimental results. Int. J. Miner. Process. 60, 213–227.

Haertel, C.J.J., Meiburg, E., 1999. Gravity Currents and Their Analysis by Direct Numerical Simulation. Fortschritt-Berichte VDI, series 7, number 366. VDI Verlag, Duesseldorf.

Landman, K.A., White, L.R., Buscall, R., 1988. The continuous-flow gravity thickener: steady state behavior. AIChE J. 34, 239–251.